

(K) Electrospun fibers ● TIMING 1 d

(i) Prepare a solution of 5% (wt/vol) PEO in water in a 20-ml glass scintillation vial. Use a small stir bar to ensure proper mixing.

□ **PAUSE POINT** The PEO will take time to dissolve into solution, so this should be done ahead of time. This solution can be stored at 4 °C for 1 year.

(ii) Add 5ml of the 5% (wt/vol) PEO (900,000 g/mol) solution into 20ml of 8% (wt/vol) silk solution to generate an aqueous solution of 6.4% (wt/vol) silk/1% (wt/vol) PEO.

□ **CRITICAL STEP** A high viscosity, because of polymer concentration and molecular weight, must be met in order for the polymer molecules to entangle and form a jet. If either the concentration or molecular weight of the polymer is too low, only beads will form. Hence, we have included PEO in this protocol to promote fiber formation by increasing the overall polymer concentration.

(iii) Slowly stir the blended solution for ~10 min at 4 °C to obtain a homogenous solution. Avoid high shear during mixing that may cause premature crystallization of the silk.

(iv) Draw up 10 ml of the silk/PEO solution into a 10 ml syringe.

(v) Attach a 16-gauge needle to the syringe.

(vi) Mount the syringe on the syringe pump.

(vii) Place a collection surface at a distance of 7–20 cm from the tip of the needle. Many surfaces can be used to collect electrospun fibers as long as they can be electrically ground. A simple surface that consists of a piece of cardboard covered with aluminum foil is sufficient.

(viii) Attach the positive voltage lead to the needle on the syringe and the ground lead to the collection surface.

(ix) Set the current to slightly above 0 A. Turn on the high voltage and syringe pump.

! **CAUTION** Electrospinning is performed at a high voltage. Be careful while the high voltage is on and do not touch charged surfaces such as the needle or positive voltage lead.

(x) Adjust the solution flow rate (0.01–0.03 ml min⁻¹), electric potential (8–15 KV) and the distance between the capillary tip and the collection screen (7–20 cm) to obtain a stable jet.

□ **CRITICAL STEP** The electric field strength is required to initiate the jet and at the lower limit bead defects are evident on the fibers. At the higher limit, silk fibers transition from a round morphology to a ribbon- shaped crosssection. It has also been noted that with an increase in voltage the fiber diameter increases because of additional mass transport with the higher electrical force. By increasing the distance between the spinneret and the collecting surface, the fiber diameter can be reduced. Finally, the flow rate should be adjusted so that the solution is constantly being supplied to the tip without allowing excess solution to drip.

(xi) Collect the silk fibers until the desired thickness is achieved.

(xii) Immerse the fiber mats in a 90% (vol/vol) methanol/water solvent for 20 min to obtain water-insoluble fiber mats.

! **CAUTION** Methanol is a hazardous and flammable substance; use proper personal safety equipment and remove all sources of ignition before use.

(xiii) Incubate the methanol-treated fiber mats in ultrapure water on a reciprocating shaker overnight to remove the PEO from the mats.

(xiv) Dry the fiber mats in a chemical fume hood.